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* * * * * Welcome to STN International * * * * *

NEWS	1		Web Page URLs for STN Seminar Schedule - N. America
NEWS	2		"Ask CAS" for self-help around the clock
NEWS	3	SEP 09	CA/CAPlus records now contain indexing from 1907 to the present
NEWS	4	AUG 05	New pricing for EUROPATFULL and PCTFULL effective August 1, 2003
NEWS	5	AUG 13	Field Availability (/FA) field enhanced in BEILSTEIN
NEWS	6	AUG 18	Data available for download as a PDF in RDISCLOSURE
NEWS	7	AUG 18	Simultaneous left and right truncation added to PASCAL
NEWS	8	AUG 18	FROSTI and KOSMET enhanced with Simultaneous Left and Right Truncation
NEWS	9	AUG 18	Simultaneous left and right truncation added to ANABSTR
NEWS	10	SEP 22	DIPPR file reloaded
NEWS	11	SEP 25	INPADOC: Legal Status data to be reloaded
NEWS	12	SEP 29	DISSABS now available on STN
NEWS	13	OCT 10	PCTFULL: Two new display fields added
NEWS	14	OCT 21	BIOSIS file reloaded and enhanced
NEWS	15	OCT 28	BIOSIS file segment of TOXCENTER reloaded and enhanced
NEWS	16	NOV 24	MSDS-CCOHS file reloaded
NEWS EXPRESS	NOVEMBER 14	CURRENT WINDOWS VERSION IS V6.0ic, CURRENT MACINTOSH VERSION IS V6.0b(ENG) AND V6.0Jb(JP), AND CURRENT DISCOVER FILE IS DATED 23 SEPTEMBER 2003	
NEWS HOURS	STN Operating Hours Plus Help Desk Availability		
NEWS INTER	General Internet Information		
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NEWS WWW	CAS World Wide Web Site (general information)		

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* * * * * STN Columbus * * * * *

FILE 'HOME' ENTERED AT 22:39:52 ON 25 NOV 2003

=>

Uploading

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Do you want to switch to the Registry File?

Choice (Y/n):

Some commands only work in certain files. For example, the EXPAND command can only be used to look at the index in a file which has an

index. Enter "HELP COMMANDS" at an arrow prompt (=>) for a list of commands which can be used in this file.

=> file registry
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
0.42	0.42

FULL ESTIMATED COST

FILE 'REGISTRY' ENTERED AT 22:41:10 ON 25 NOV 2003
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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 24 NOV 2003 HIGHEST RN 620531-14-8
DICTIONARY FILE UPDATES: 24 NOV 2003 HIGHEST RN 620531-14-8

TSCA INFORMATION NOW CURRENT THROUGH JULY 14, 2003

Please note that search-term pricing does apply when conducting SmartSELECT searches.

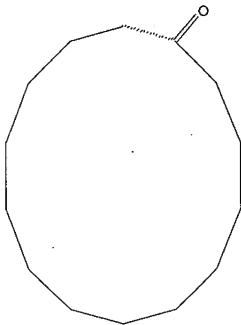
Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNnote 27, Searching Properties in the CAS Registry File, for complete details:
<http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf>

=>
Uploading 10015975.str

L1 STRUCTURE UPLOADED

=> d l1
L1 HAS NO ANSWERS
L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1 sss sam
SAMPLE SEARCH INITIATED 22:41:32 FILE 'REGISTRY'

SAMPLE SCREEN SEARCH COMPLETED - 314465 TO ITERATE

0.3% PROCESSED 1000 ITERATIONS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.01

23 ANSWERS

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
BATCH **INCOMPLETE**
PROJECTED ITERATIONS: EXCEEDS 1000000
PROJECTED ANSWERS: EXCEEDS 139557

L2 23 SEA SSS SAM L1

=> s l1 sss full
FULL SEARCH INITIATED 22:41:43 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - >1,000,000 TO ITERATE

< 6.4% PROCESSED 400000 ITERATIONS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.06

2137 ANSWERS

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
BATCH **INCOMPLETE**
PROJECTED ITERATIONS: EXCEEDS 1000000
PROJECTED ANSWERS: EXCEEDS 33080

L3 2137 SEA SSS FUL L1

=> file caplus
COST IN U.S. DOLLARS
FULL ESTIMATED COST

SINCE FILE	TOTAL
ENTRY	SESSION
148.15	148.57

FILE 'CAPLUS' ENTERED AT 22:41:55 ON 25 NOV 2003
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FILE COVERS 1907 - 25 Nov 2003 VOL 139 ISS 22
FILE LAST UPDATED: 24 Nov 2003 (20031124/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l3 and (musk or perfum? or fragran?)

439 L3
2399 MUSK
682 MUSKS
2539 MUSK
(MUSK OR MUSKS)

29913 PERFUM?
11729 FRAGRAN?

L4 0 L3 AND (MUSK OR PERFUM? OR FRAGRAN?)

=> s 13

L5 439 L3

=> s 15 and (odor? or scent? or aroma)

75705 ODOR?

2258 SCENT?

15422 AROMA

1487 AROMAS

15957 AROMA

(AROMA OR AROMAS)

L6 1 L5 AND (ODOR? OR SCENT? OR AROMA)

=> d 16 hitstr, ibib, iabs

L6 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2003 ACS on STN

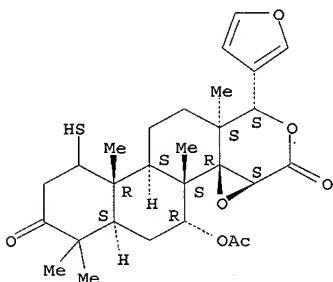
IT 548776-47-2D, derivs. 548776-48-3

RL: NPO (Natural product occurrence); PAC (Pharmacological activity); THU (Therapeutic use); BIOL (Biological study); OCCU (Occurrence); USES (Uses) (traditionally-used antimalarials from the Meliaceae)

RN 548776-47-2 CAPLUS

CN Oxireno[c]phenanthro[1,2-d]pyran-3,8(3aH,4bH)-dione, 5-(acetyloxy)-1-(3-furanyl)dodecahydro-10-mercapto-4b,7,7,10a,12a-pentamethyl-, (1S,3aS,4aR,4bS,5R,6aS,10aR,10bS,12aS)- (9CI) (CA INDEX NAME)

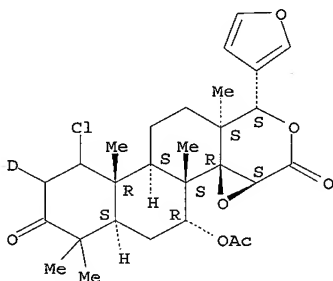
Absolute stereochemistry.



RN 548776-48-3 CAPLUS

CN Oxireno[c]phenanthro[1,2-d]pyran-3,8(3aH,4bH)-dione-9-d, 5-(acetyloxy)-10-chloro-1-(3-furanyl)dodecahydro-4b,7,7,10a,12a-pentamethyl-, (1S,3aS,4aR,4bS,5R,6aS,10aR,10bS,12aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



ACCESSION NUMBER:

2003:43649 CAPLUS

DOCUMENT NUMBER:

139:62350

TITLE:

Traditionally-used antimalarials from the Meliaceae

AUTHOR(S):

Omar, S.; Zhang, J.; MacKinnon, S.; Leaman, D.; Durst, T.; Philogene, B. J. R.; Arnason, J. T.; Sanchez-Vindas, P. E.; Poveda, L.; Tamez, P. A.; Pezzuto, J. M.

CORPORATE SOURCE:

Ottawa-Carleton Institutes of Biology and Chemistry, University of Ottawa, Ottawa, ON, K1N 6N5, Can.

SOURCE:

Current Topics in Medicinal Chemistry (Hilversum, Netherlands) (2003), 3(2), 133-139

PUBLISHER:

CODEN: CTMCCL; ISSN: 1568-0266

DOCUMENT TYPE:

Bentham Science Publishers Ltd.

LANGUAGE:

Journal; General Review

ABSTRACT:

English

A review. A quant. ethnobotanical approach to antimalarial drug discovery led to the identification of *Lansium domesticum* Corr. Ser. (Meliaceae) as an important antimalarial used by Kenyah Dyak healers in Indonesian Borneo. Triterpenoid lansiolidides with antimalarial activity were isolated from the bark and shown to have activity in both in vitro bioassays with *Plasmodium falciparum*, and in mice infected with *P. berghei*. A survey of African and tropical American Meliaceae led to further development of the limonoid gedunin from the traditionally used medicinal plants, tropical cedar, *Cedrela odorata* L., and neem, *Azadirachta indica* A. Juss. Gedunin has significant in vitro activity but initially showed poor in vivo activity. In vivo activity was improved by (1) incorporation into an easy to absorb suspension, (2) prepn. of a more stable compd., 7-methoxygedunin; and (3) synergism with dillapiol, a cytochrome P 450 3A4 inhibitor. The results show the potential for both antimalarial drug and phytomedicine development from traditionally used plants.

REFERENCE COUNT:

20

THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> s 15 and smell

4117 SMELL

502 SMELLS

4515 SMELL

(SMELL OR SMELLS)

L7

0 L5 AND SMELL

=> s 15 and (musk or perfum? or fragran?)

2399 MUSK

682 MUSKS

2539 MUSK

(MUSK OR MUSKS)
 29913 PERFUM?
 11729 FRAGRAN?
 L8 0 L5 AND (MUSK OR PERFUM? OR FRAGRAN?)
 => s (cyclotetradecanone or cyclotetradecenone)
 82 CYCLOTETRADECANONE
 2 CYCLOTETRADECANONES
 83 CYCLOTETRADECANONE
 (CYCLOTETRADECANONE OR CYCLOTETRADECANONES)
 9 CYCLOTETRADECENONE
 3 CYCLOTETRADECENONES
 9 CYCLOTETRADECENONE
 (CYCLOTETRADECENONE OR CYCLOTETRADECENONES)
 L9 88 (CYCLOTETRADECANONE OR CYCLOTETRADECENONE)

=> s l9 and (musk or perfum? or fragran?)
 2399 MUSK
 682 MUSKS
 2539 MUSK
 (MUSK OR MUSKS)
 29913 PERFUM?
 11729 FRAGRAN?
 L10 16 L9 AND (MUSK OR PERFUM? OR FRAGRAN?)

=> d l10 ibib, iabs 1-16

L10 ANSWER 1 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN
 ACCESSION NUMBER: 2003:356108 CAPLUS
 DOCUMENT NUMBER: 138:358213
 TITLE: Preparation and organoleptic uses of macrocyclic musks
 INVENTOR(S): Mookherjee, Braja Dulal; Narula, Anubhav P. S.; Monteleone, Michael G.; Trenkle, Robert W.
 PATENT ASSIGNEE(S): International Flavors & Fragrances Inc., USA
 SOURCE: Eur. Pat. Appl., 8 pp.
 CODEN: EPXXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1308497	A2	20030507	EP 2002-257554	20021031
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK				
US 2003087797	A1	20030508	US 2001-15975	20011101
PRIORITY APPLN. INFO.:			US 2001-15975	A 20011101

ABSTRACT:

Mixts. consisting essentially of oxomacrocyclic compds. contg. >40% .DELTA.3-cyclotetradecen-1-one and >40% cyclotetradecanone and <5% .DELTA.2-cyclotetradecen-1-one, are useful as perfumery components. In addn. a chemoselective catalytic hydrogenation process for prepn. of compds. by hydrogenating mixts. contg. significant concns. (>15%) of .DELTA.2-cyclotetradecen-1-one. The oxocarbocyclic compd.-contg. mixts. have advantageous musk aroma imparting, augmenting and enhancing ***perfumery*** properties causing the mixts. to be useful in perfume compns., perfumed articles and perfumed polymers, including particles and fibers produced therefrom. Thus, a mixt. of .DELTA.3-cyclotetradecen-1-one and .DELTA.2-cyclotetradecen-1-one was prepd. by the pyrolysis of 2-hydroxycyclotetradecan-1-one in the presence of Al2O3 particles. The resulting product had a musk aroma with waxy and oily top-notes and undernotes. The product was used in various perfume formulations.

L10 ANSWER 2 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN
 ACCESSION NUMBER: 2002:944465 CAPLUS
 DOCUMENT NUMBER: 138:28944
 TITLE: Skin-care products containing melanin inhibitors
 INVENTOR(S): Matsuda, Hiroyuki; Yamamoto, Kenichi; Tamai, Eiko;
 Hagiwara, Toshimitsu; Yagi, Misao; Watanabe, Sinya;
 Kumamoto, Hiroyasu
 PATENT ASSIGNEE(S): Takasago International Corporation, Japan
 SOURCE: Eur. Pat. Appl., 23 pp.
 CODEN: EPXXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1264594	A2	20021211	EP 2002-291413	20020607
EP 1264594	A3	20030305		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
JP 2002363071	A2	20021218	JP 2001-173655	20010608
JP 2003119128	A2	20030423	JP 2001-315378	20011012
US 2003049213	A1	20030313	US 2002-164702	20020610
PRIORITY APPLN. INFO.:			JP 2001-173655	A 20010608
			JP 2001-315378	A 20011012

OTHER SOURCE(S): MARPAT 138:28944

ABSTRACT:

The invention relates to a melanin synthesis inhibitor compn. contg. at least one macrocyclic compd. such as cyclotetradecanone, cyclopentadecanone, 4-cyclopentadecenone. Thus, a cosmetic lotion was prepd. from 2 phases; the oil phase contained 5-cyclohexadecenol 0.01, EtOH 20.0, hydrogenated ethoxylated castor oil 0.05, Me p-hydroxybenzoate 0.1, and ***perfume*** 0.1%; the aq. phase comprised glycerin 10.0, 1,3-butylene glycol 5.0, and water qs to 100%. The 2 phases were mixed to give a cosmetic lotion which had a skin-lightening effect and good storage stability. The effectiveness of this compd. in inhibiting melanin synthesis was demonstrated. The preps. of the compds. of the invention, e.g, cycloalkanones, cycloalkenols, are given.

L10 ANSWER 3 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2002:140194 CAPLUS
 DOCUMENT NUMBER: 137:20088
 TITLE: A novel, short and repeatable two-carbon ring expansion reaction by thermo-isomerization: easy synthesis of macrocyclic ketones
 AUTHOR(S): Nagel, Matthias; Hansen, Hans-Jurgen; Frater, Georg
 CORPORATE SOURCE: Organisch-chemisches Institut, Universitat Zuerich, Zurich, CH 8057, Switz.
 SOURCE: Synlett (2002), (2), 275-279
 CODEN: SYNLES; ISSN: 0936-5214
 PUBLISHER: Georg Thieme Verlag
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 137:20088

ABSTRACT:

A novel two-carbon ring enlargement procedure, in which medium- and large-ring 1-vinylcycloalkanols are thermo-isomerized in a flow reactor system at temps. of 600.degree.C to about 650.degree.C, produces the isomeric ring-expanded cycloalkanones directly and efficiently. This two-step ring expansion protocol can easily be applied several times successively. For e.g., the musk odorant cyclopentadecanone (Exaltone) is prepd. from cycloundecanone in two

repetitive cycles. Thermo-isomerization of the corresponding ethynylcycloalkanols gives in moderate yields the bishomologous .alpha.,.beta.-unsatd. macrocyclic (E)-2-cycloalkenones. A reaction mechanism via alkyl hydroxyallyl biradical intermediates is proposed.

REFERENCE COUNT: 95 THERE ARE 95 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 4 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1997:809593 CAPLUS

DOCUMENT NUMBER: 128:92973

TITLE: Dependence of intensity of **musk** odor on the energy gap between frontier molecular orbitals

AUTHOR(S): Gorbachov, M. Yu.

CORPORATE SOURCE: INSTITUTE OF CHEMISTRY OF AS, Chisinau, MD 2028, Moldova

SOURCE: Special Publication - Royal Society of Chemistry (1997), 214 (Flavours and Fragrances), 48-53
CODEN: SROCDQ; ISSN: 0260-6291

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

ABSTRACT:

It is clear that a term .zeta. can be used to predict the intensity of ***musk*** odor compds. contg. 2 active mol. fragments provided that there are no bulky substituents near the atoms of these fragment. The effect of different bulky groups on **musk** odor intensity will be considered.

REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 5 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1992:151224 CAPLUS

DOCUMENT NUMBER: 116:151224

TITLE: Preparation of bicyclo[10.2.0]tetradecan-13-one as **fragrance** and its dichlorinated intermediate

INVENTOR(S): Evers, William J.; Heinsohn, Howard H., Jr.

PATENT ASSIGNEE(S): International Flavors and Fragrances Inc., USA

SOURCE: U.S., 15 pp.
CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

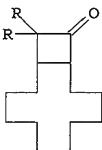
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5077274	A	19911231	US 1990-596506	19901012
EP 480126	A1	19920415	EP 1991-109330	19910607

R: CH, DE, ES, FR, GB, LI, NL

PRIORITY APPLN. INFO.: US 1990-596506 19901012

OTHER SOURCE(S): MARPAT 116:151224

GRAPHIC IMAGE:



ABSTRACT:

The title compds. I (R = H, Cl), are prepd. Cyclododecene was added to Cl₃CCOCl at 20-24.degree. and refluxed at 42.degree. for 35 h to give I (R = Cl) which at 25-29.degree. was reacted with Zn dust followed by NH₄Cl and refluxed at 64.degree. to give I (R = H) (II). II imparts a vertiver formulation musky and earthy undertones and minty, green and camphoreaceous topnotes.

L10 ANSWER 6 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1991:489815 CAPLUS

DOCUMENT NUMBER: 115:89815

TITLE: Origin of musk fragrance activity:
the electron-topologic approach

AUTHOR(S): Bersuker, I. B.; Dimoglo, A. S.; Gorbachov, M. Yu.;
Vlad, P. F.; Pesaro, M.

CORPORATE SOURCE: Inst. Chem., Kishinev, 277028, USSR

SOURCE: New Journal of Chemistry (1991), 15(5), 307-20

CODEN: NJCHE5; ISSN: 0398-9836

DOCUMENT TYPE: Journal

LANGUAGE: English

ABSTRACT:

Based on the electron-topol. approach to the QSAR problem worked out earlier by some of the authors, the rules of musk fragrance activity are revealed and discussed in detail. It is shown that the mol. feature responsible for the musk odor can be represented by an appropriate electron-topol. matrix that includes 2 independent mol. fragments with special electronic and topl. characteristics. The 1st consist of a polar group (CO, NO, CN) whose electroneg. atom is situated sym. and at a distance of 6.7 .ANG. with respect to 2 Me (or methylene) groups, the distance between them being 2.5 .ANG.. The 2nd fragment includes 2 other Me (or methylene) groups situated at a distance of 5.5 .ANG. from each other. These 2 features are necessary conditions for musk odor, the sufficient ones including mainly the requirement that there are no bulky substituents close to the functional group making the latter sterically unaccessible. There are 362 org. compds. from different classes including macrocycles, nitrobenzene, indane, and hydronaphthalene derivs. that are analyzed in view of the musk revealed in this work, and only a few of them (7 active compds. and 8 inactive ones) apparently don't obey these rules; the Bayes probability of prediction of ***musk*** odor with these rules is P = 0.96. The origin of the discrepancies between the musk rules and exptl. data are discussed, and a hypothesis of dimers activity is suggested.

L10 ANSWER 7 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1989:120987 CAPLUS

DOCUMENT NUMBER: 110:120987

TITLE: Studies on the volatile components of secretion from
Chinese civet (Verrucula indica Desmarest)

AUTHOR(S): Ding, Desheng; Yu, Duiyi; Zhu, Zemin; Zhang, Chengzeng;
Fu, Yiyuan

CORPORATE SOURCE: ., Minist. Light Ind., Shanghai, Peop. Rep. China

SOURCE: Developments in Food Science (1988), 18(Flavors

Fragrances), 587-600

CODEN: DFSCDX; ISSN: 0167-4501

DOCUMENT TYPE: Journal

LANGUAGE: English

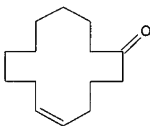
ABSTRACT:

The volatile components of the Chinese civet were obtained by microsteam distn. of both fresh and aged secretion. Using a combination of GC, GC/MS, GC-FTIR and other techniques, the volatile constituents of Chinese civet were examd. Thus far, more than 80 components were isolated and identified, with 60 of them characterized for the first time. In the neutral fraction there exists a series of macrocyclic ketones such as civetone and muscone. Obviously, the presence of macrocyclic compds. is indicative of the high value of Chinese

civet in the perfumery industry.

L10 ANSWER 8 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN
ACCESSION NUMBER: 1980:604157 CAPLUS
DOCUMENT NUMBER: 93:204157
TITLE: 5-cis-Cyclotetradecen-1-one
PATENT ASSIGNEE(S): Takasago Perfumery Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 55066534	A2	19800520	JP 1978-139680	19781113
JP 57042614	B4	19820909		
PRIORITY APPLN. INFO.:			JP 1978-139680	19781113
GRAPHIC IMAGE:				



I

ABSTRACT:

The title compd I was prepd. by heating 1-vinyl-3-cis-cyclododecen-1-ol (II) with alkali metals or alkali hydrides in Me₂NAC, Et₂NCOEt, N-methylpyridone, or N-methylcaprolactam. Thus, 2.4 g NaH was added to 10 g II in Me₂NAC at room temp. and the mixt. kept 17 h at 80.degree. to give 10 g product contg. 56% I and 44% 4-vinyl-1-cyclododecanone. I is useful as **musk**
perfume

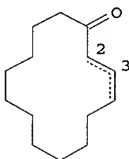
L10 ANSWER 9 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN
ACCESSION NUMBER: 1980:203426 CAPLUS
DOCUMENT NUMBER: 92:203426
TITLE: 2- and 3-Cyclotetradecen-1-ones as bitterness depressants
INVENTOR(S): Mookherjee, Braja D.; Trenkle, Robert W.; Vock, Manfred H.; Luccarelli, Domenick, Jr.; Schmitt, Frederick L.; Stork, Gilbert; MacDonald, Timothy; Liberman, Arthur L.
PATENT ASSIGNEE(S): International Flavors and Fragrances Inc., USA
SOURCE: U.S., 19 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4183965	A	19800115	US 1978-973093	19781226
US 4215006	A	19800729	US 1979-52830	19790627
US 4224352	A	19800923	US 1979-52829	19790627
US 4282274	A	19810804	US 1979-52668	19790627

PRIORITY APPLN. INFO.:
GRAPHIC IMAGE:

US 1978-973093

19781226



I

ABSTRACT:

2- And 3-cyclotradecen-1-ones (I) are prepd. and have a sweet, brown sugar and musky aroma, useful for food flavoring or bitterness depressants for foods or used in perfumes to impart a sweet, musk aroma and exaltone-like scorched linen, waxy rooty nuances. Treating 1-(1-cyclododecenyl)pyrrolidine [25769-05-5] with Et propiolate [623-47-2] gave, after gas chromatog., I (.DELTA.3, cis isomer) (II) [73639-27-7], I (.DELTA.2, trans isomer) (III) [73639-28-8], I (.DELTA.3, trans isomer) (IV) [73639-29-9] and cyclotetradecanone [3603-99-4]. Examples were given for addn. of II, III, and IV mixts. to food flavor, detergent, ***perfume***, and soap compns.

L10 ANSWER 10 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1965:446147 CAPLUS
DOCUMENT NUMBER: 63:46147
ORIGINAL REFERENCE NO.: 63:8320f-h,8321a-g
TITLE: New alicyclic enamines and the method of their preparation
INVENTOR(S): Brannock, Kent C.
PATENT ASSIGNEE(S): Eastman Kodak Co.
SOURCE: 21 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1397172		19650430	FR	

PRIORITY APPLN. INFO.: US 19621031
GRAPHIC IMAGE: For diagram(s), see printed CA Issue.

ABSTRACT:

The title compds. (I), in which R is an alkyl group, Q represents nonmetallic elements to form a satd. heterocyclic ring, X is H or CO₂R, and Y is a methylene chain of 3 to 14 C atoms, are prepd. CH.tplbond.CO₂Me (15 g.) is added (5 min.) to 27 g. N-(1-cyclopent-1-enyl)piperidine (II) in 75 ml. MeCN, causing the temp. to rise to 85.degree.. After the temp. dropped to 34.degree., the mixt. is distd. at 120.degree./90 mm., giving Me 2-(1-piperidyl)-1,6-cycloheptadiene-1-carboxylate which treated with 20 ml. concd. HCl in 100 ml. H₂O 5 hrs. at 20.degree. and the mixt. extd. with Et₂O, gives 27% Me 2-hydroxy-1,6-cycloheptadiene-1-carboxylate (III), b₁ 5 71-6.degree., n₂₀D 1.5236. With N-(1-cyclopentenyl)pyrrolidine (IV) in lieu of II the Me 2-(1-pyrrolidinyl) analog is formed and on hydrolysis with HCl it also gives III. III has a strong odor of o-HOC₆H₄CO₂Me, it forms chelates with Cu and Co ions, it absorbs 1 mole H on hydrogenation, and on sapon. and decarboxylation it gives an overall yield of 74% cycloheptanone. Adding 28.4

g. (.tplbond.CCO2Me)2 (10 min.) to 30.2 g. IV in 150 ml. Et2O at 25-35.degree., keeping the mixt. 0.5 hr., and evapg. the Et2O give 71% di-Me 3-(1-pyrrolidinyl)-2,7-cycloheptadiene-1,2-dicarboxylate (V), m. 147-8.degree., which treated with 10 ml. concd. HCl in 40 ml. H2O gives 49% di-Me 3-hydroxy-2,7-cycloheptadiene-1,2-carboxylate, b0.4-0.5 101-6.degree.. Adding (10 min.) 28.4 g. (.tplbond.CCO2Me)2 to 30.2 g. N(1-cyclohex-1-enyl)pyrrolidine in 150 ml. Et2O at 25-35.degree., keeping the mixt. 10 min., adding 150 ml. C5H12, and cooling give 70% di-Me 1-(1-pyrrolidinyl)bicyclo [4.2.0]-7-octene-7,8-dicarboxylate (VI), m. 77-81.degree.. VI is quite unstable toward heat. VI (14.7 g.) treated 3 days with 10 ml. HCl in 40 ml. H2O gives 2.5 g. di-Me 3-hydroxy-2,8-cyclooctadiene-1,2-dicarboxylate, b1 123-6.degree., m. 74-5.degree.. VI (37 g.) heated 18 hrs. on a water bath at 85.degree. and the mixt. extd. with Et2O gives 4 g. di-Me 3-(1-pyrrolidinyl)-2,8-cyclooctadiene-1,2-dicarboxylate, m. 141-2.degree., which, hydrolyzed, gives di-Me 3-hydroxy-2,8-cyclooctadiene-1,2-dicarboxylate. Similarly, 3.86 g. N-(1-cyclooctenyl)piperidine in 25 ml. Et2O and 3.4 g. (.tplbond.CCO2Et)2 in 25 ml. Et2O give 61% di-Et 3-piperidine-2,10-cyclodecadiene-1,2-dicarboxylate, m. 91-2.degree.. Adding (5 min.) 3.3 g. N-(1-cycloheptenyl)pyrrolidine (VII) in 25 ml. Et2O to 2.8 g. (.tplbond.CCO2Me)2 at 30-5.degree. and keeping the mixt. 0.5 hr. at 20.degree. gives 93% di-Me 3-(1-pyrrolidinyl)-2,9-cyclononadiene-1,2-dicarboxylate, m. 109.5-10.5.degree.. Hydrogenating 74 g. V in 500 ml. AcOH in the presence of 2 g. PtO2 at 20.degree. and 2.8 atm. H, dissolving the residue of the filtered and evapg. soln. in 250 ml. H2O and 250 ml. MeOH contg. 75 g. KOH, heating the mixt. 23 hrs. at 100.degree., dilg. the concd. soln. with 150 ml. H2O, and acidifying it with HCl gives 73% 2-cycloheptene-1,2-dicarboxylic acid, m. 168-70.degree.. Its Na salt hydrogenated with Raney Ni and acidified gives 95% cis-cycloheptane-1,2-dicarboxylic acid, m. 130-1.degree.. The latter readily rearranges to the trans isomer, m. 156-7.degree., on heating with dil. H2SO4. Addn. (10 min.) of 28.4 g. (.tplbond.CCO2Me)2 to 27.4 g. 1-(2,5-dihydro-3-furyl)pyrrolidine in 150 ml. Et2O at 25-35.degree. gives 17% di-Me 1-(1-pyrrolidinyl)-6-oxa-1,3-cycloheptadiene-2,3-dicarboxylate, m. 162-3.degree.. Addn. of 0.2 mole (.tplbond.CCO2Me)2 to 0.2 mole N-(1-cyclododecenyl)pyrrolidine gives 91% di-Me 3-(1-pyrrolidinyl)-2,14-cyclotetradecadiene-1,2-dicarboxylate, m. 94-5.degree.. Adding 30.2 g. CH.tplbond.CCO2Me to 64 g. N-(1-cyclooctenyl)pyrrolidine in 200 ml. Et2O at 35.degree. and evapg. the Et2O give 100% Me 2-(1-pyrrolidinyl)-1,9-cyclodecadiene-1-carboxylate, m. 102-3.degree., which hydrolyzed with HCl gives 50% partially enolized Me 10-oxo-2-cyclodecene-1-carboxylate, b0.8 103-8.degree., n20D 1.4953. On hydrogenation, sapon., and decarboxylation it gives 76% cyclodecanone. Addn. of 1.42 g. (.tplbond.CCO2Me)2 to 1.67 g. N-(1-cyclohexenyl)morpholine in 10 ml. Et2O at below 35.degree., evapn. of the Et2O, and heating the residue 1.5 hrs. at 100.degree. give 29% di-Me 3-morpholino-2,8-cyclooctadiene-1,2-dicarboxylate, m. 216-17.degree.. Similarly, 5 g. (.tplbond.CCO2Me)2 and 8.5 g. N-(1-cyclododecenyl)pyrrolidine in 25 ml. Et2O at 30-5.degree. gives 90.5% di-Me 3-(1-pyrrolidinyl)-2,14-cyclotetradecadiene-1,2-dicarboxylate, m. 94-5.degree., which treated with 50 ml. HCl in 200 ml. H2O gives 83% di-Me 3-hydroxy-2,14-cyclotetradecadiene-1,2-dicarboxylate (VIII), m. 73-4.degree.. Hydrogenation of 43 g. VIII in MeOH over 5% Pd-C gives 84% di-Me 3-oxocyclotetradecane-1,2-dicarboxylate, m. 93.degree.. Condensation of 20 g. VII in 50 ml. Et2O with 10.5 g. CH.tplbond.CCO2Me at 20-30.degree. gives 20 g. Me 2-(1-pyrrolidinyl)-1,8-cyclononadiene-1-carboxylate, m. 82-7.degree., which hydrogenated over 5% Pd-C in AcOH gives 13.5 g. Me 2-(1-pyrrolidinyl)-1-cyclononene-1-carboxylate, m. 84-5.degree.. The latter sapon. with 25% NaOH and distd. gives cyclononane. Adding dropwise 8.4 g. CH.tplbond.CCO2Me in 50 ml. Et2O to 23.5 g. N-(1-cyclododecenyl)pyrrolidine in boiling C6H14 gives 82% Me 2-(1-pyrrolidinyl)-1,13-cyclotetradecadiene-1-carboxylate, m. 60-2.degree., which with HCl at 50-60.degree. gives 77% Me 2-hydroxy-1,13-cyclotetradecadiene-1-carboxylate, m. 48-9.degree.. The latter (13.5 g.) in 100 ml. MeOH is passed through an Al2O3 column, the soln. treated with C, and hydrogenated over 5% Pd-Al2O3 at 20.degree. and 2.8 atm. to give 93% Me 2-oxocyclotetradecane-1-carboxylate, which sapon. with 50% NaOH in 100 ml. H2O and 50 ml. MeOH 72 hrs. at 20.degree. gives 66% cyclotetradecanone, 51-2.degree.. I are useful as intermediates for perfumes and the dicarboxylic acids for the prepn. of polymeric esters.

L10 ANSWER 11 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN
ACCESSION NUMBER: 1962:2327 CAPLUS
DOCUMENT NUMBER: 56:2327
ORIGINAL REFERENCE NO.: 56:450f-i,451a
TITLE: A new method of synthesis of macrocyclic ketones with musk odor
AUTHOR(S): Belen'kii, L. I.; Taits, S. Z.; Gol'dfarb, Ya. L.
CORPORATE SOURCE: N. D. Zelinskii Inst. Org. Chem., Moscow
SOURCE: Doklady Akademii Nauk SSSR (1961), 139, 1356-8
CODEN: DANKAS; ISSN: 0002-3264

DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GRAPHIC IMAGE: For diagram(s), see printed CA Issue.

ABSTRACT:
To 0.3 mole AlCl₃.Et₂O (b0.5 100-3.degree., b1 108-10.degree., b3 124-5.degree., b4 128-9.degree., b12 150.degree. with decompn.; m. about 35.degree.) in 300 ml. Et₂O was added an equimolar amt. of H₂O over 10 hrs., the addn. being performed by means of a special adapter from which H₂O was transferred into the reaction mixt. by refluxing solvent; after complete dissoln. of the added H₂O, 1.5 l. CHCl₃ was added, Et₂O distd., and the refluxing soln. treated with the desired thienylalkanoyl chloride at the rate of 0.015-0.3 .times. 10-4 mole per hr. to yield the following
.alpha.-cyclothien-1-ones (I) (n shown): 9, b1 149-52.degree., m. 40-1.degree., 53.67%; 10, b0.05 128-32.degree., m. 44.5-6.5.degree., 63.5%; 11, b0.5 162-5.degree., m. 31-2% 62.5.degree., 12, b0.005 157.degree., n20D 1.5448, d20 1.0480, 60%. Their semicarbazones m. 188.5-9.5.degree., 194-6.degree., 214-15.degree., and 225-6.5.degree., resp. Stirred with Raney Ni in EtOH-Me₂CO these gave: cyclotetradecanone 75%; cyclohexadecanone m. 62.5-64.degree. (semicarbazone m. 178-80.5.degree.); dihydrocivetone m. 61-3.degree. (semicarbazone m. 189.5-91.degree.).

L10 ANSWER 12 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN
ACCESSION NUMBER: 1957:71655 CAPLUS
DOCUMENT NUMBER: 51:71655
ORIGINAL REFERENCE NO.: 51:12970i,12971a-b
TITLE: Civetone and its homologs
INVENTOR(S): Blomquist, Alfred T.; Wolinsky, Joseph
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2790005	---	19570423	US	-----

ABSTRACT:
Civetone homologs with a strong musk odor may be produced from the corresponding diketones. 1,9-Cyclohexadecanedione (0.503 g.) in AcOH hydrogenated with 70 mg. pre-reduced Adams catalyst until 104% of 1 equiv. of H had been absorbed, and the resulting solid extd. with hexane and crystd. at 0-5.degree. gave 0.22 g. 9-hydroxycyclohexadecanone (I); another 0.09 g. was recovered from the hexane by elution in a Magnesol-Celite column with CHCl₃-C₆H₆. I, m. 76-77.degree., (170 mg.) and 1.5 g. KHSO₄ heated 5 min. at 250.degree., then cooled, extd. with pentane, and the pentane evapd. gave 145 mg. 8-cyclohexadecen-1-one, m. 17-22.degree.. Similarly, 0.81 g. 1,10-cyclooctadecanedione reduced, dissolved in hexane, placed on an alumina column, and eluted with a hexane-heptane mixt. gave 0.23 g. starting material; elution with hexane-C₆H₆ and recrystn. gave 0.23 g. 10-hydroxycyclooctadecanone (II), m. 80-1.degree., and final elution with CHCl₃ yielded 0.16 g. 1,10-cyclooctadecanediol. II (155 mg.) and 2 g. KHSO₄ gave 9-cyclooctadecen-1-one, m. 40-1.degree. (sublimes).

L10 ANSWER 13 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1948:17399 CAPLUS
DOCUMENT NUMBER: 42:17399
ORIGINAL REFERENCE NO.: 42:3741h-i,3742a-b
TITLE: Syntheses of macrocyclic products of a musk
odor. III. Cyclic acyloins
AUTHOR(S): Stoll, M.; Rouve, A.
CORPORATE SOURCE: Maison Firmenich & Cie., Geneve, Switz.
SOURCE: Helvetica Chimica Acta (1947), 30, 1822-36
CODEN: HCACAV; ISSN: 0018-019X
DOCUMENT TYPE: Journal
LANGUAGE: French
ABSTRACT:

Several cyclic acyloins have been prep'd. by the method of the preceding abstr. Little care need be taken to employ either extreme diln. or slow addn. to attain good yields. For the compds. prep'd. are given the yield (where stated), m.p., d₄55, n_D55, and m.p. of the oxime: azelaoin (2-ketocyclononanol) (I), 18%, 40-2.degree., 1.0340, 1.4767, 117-18.degree.; sebacoin (2-ketocyclodecanol), 45.5%, 38-9.degree., 1.0200, 1.4794, 100-2.degree.; 2-ketocyclohendecanol, 30-3.degree., 1.0071, 1.4797, 119-20.degree.; 2-ketocyclododecanol, 76.2%, 78-9.degree., 0.9959, 1.4803, 131-1.5.degree.; brassyloin (2-ketocyclotridecanol), 67.5%, 45-6.degree., 0.9840, 1.4786, 98-9.degree.; 2-ketocyclotetradecanol, 79%, 84-5.degree., 0.9723, 1.4771, 123-4.degree.; exaltoin (2-ketocyclopentadecanol), 77%, 57-8.degree., 0.9632, 1.4765, 110-11.degree.; thapsoin (2-ketocyclohexadecanol), 84%, 58-9.degree., 0.9498, 1.4756, 110-11.degree.; 2-ketocycloheptadecanol, 85%, 53-4.degree., 0.9410, 1.4740, 119-20.degree.; 2-ketocyclooctadecanol, 59-60.degree., 0.9330, 1.4720, 112-12.5.degree.; and 1-methyl-2-keto-3-cyclopentadecanol, m. below 0.degree., d₄17.5 0.9743, n_D17 1.4874. These acyloins are sensitive to O, passing through the cyclic diketone to the acyclic diacid; also a doubling of the mol. occurs. Their odor resembles that of the cyclanones contg. large rings. In the prepn. of I, a by-product is cyclooctadecane-1,9-diol-2,10-dione, b₀.12 200-30.degree..

L10 ANSWER 14 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1929:9680 CAPLUS
DOCUMENT NUMBER: 23:9680
ORIGINAL REFERENCE NO.: 23:1110h-i,1111a-c
TITLE: Carbon rings. XIII. Oxidation of the 13-to 17-membered monocyclic ketones with Caro's acid to the 14-to 18-membered lactones
AUTHOR(S): Ruzicka, L.; Stoll, M.
SOURCE: Helvetica Chimica Acta (1928), 11, 1159-73
CODEN: HCACAV; ISSN: 0018-019X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GRAPHIC IMAGE: For diagram(s), see printed CA Issue.
ABSTRACT:

cf. C. A. 22, 4482. Angelica oil contains the lac-tone (I) of pentadecan-15-ol-1-acid, and musk-seed oil the lactone (II) of ambrettolic acid: Kerschbaum (C. A. 21, 2118) does not state the properties of I. Ambrettolide is described as a liquid b₁₆ 185-190.degree., and d₂₀ 0.938. Its C content is 1-1.5% above the theoretical. Kerschbaum obtains by catalytic reduction of ambrettolide the lactone of hexadecan-16-ol-1-acid, a thin liquid b. 175-180.degree.. The constitution of this acid is proven by oxidation to the HO acid. He describes a synthetic mode of formation of I. which has a ***** fragrance but no further properties are given. The evidence that the musk fragrance comes from the lactone lies in the loss of the odor after sapon. The exact proof of relation between the no. of ring members and odor necessitates knowledge of the pure lactone. The pure lactone originates through the reaction of Caro's acid on the ketone. The authors believe that 3 classes of compds. arise, a simple peroxide, polymeric peroxide and the lactone. Bacyer and Villager now assume the simple

peroxide to lie in the lactone. The lactone is isolated by means of menthol or camphor. With exaltone (cyclopentadecanone) the direction of the reaction of Caro's acid on a high-membered ketone is investigated. In petroleum ether below 20.degree. there is a slow reaction, at 30.degree. the lactone and polymeric peroxide are formed, and at 60-65.degree. a 50% yield of rather pure lactone is given. In all cases arises a mixt. of etholide, which, purified by sapon., gives the pentadecan-15-ol-1-acid. The lactone of the acid, sapond. by warm alc. alkali, forms the HO acid, which on oxidation yields tridecane-1,13-dicarboxylic acid. In an analogous manner and with similar results the 13-, 14-, 16- and 17-membered ketones are transformed to lactones. The phys. properties of this homologous series of 5 high-membered lactones run in a regular course. The HO acids show a homogeneous oscillation of m. p. like the other series of aliphatic compds.

L10 ANSWER 15 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1928:37634 CAPLUS

DOCUMENT NUMBER: 22:37634

ORIGINAL REFERENCE NO.: 22:4483g-i,4484a-g

TITLE: Carbon rings. XII. The preparation of methylated 14-, 15- and 17-membered cyclic ketones

AUTHOR(S): Ruzicka, L.; Schinz, H.; Pfeiffer, M.

CORPORATE SOURCE: Utrecht Univ.

SOURCE: Helvetica Chimica Acta (1928), 11, 686-700

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

ABSTRACT:

Since muscone is a 1-methylcyclopentadecan-3-one, attempts were made to prep. similar cyclic ketones with the Me group in a position 1, 2, 3 and 4 to the CO group and to compare the musk odors of such compds. The method of prepn. consisted in heating the Th or Y salts of polymethylenedicarboxylic acids and it was found that only those compds. with the Me group in position 3 or 4 yielded cyclic ketones. 1,3-Dimethylcyclotridecan-2-one (5.4 g.) does not have a musk odor and was prepd. by destructive distn. of 173 g. of the Th salt of tetradecane-2,13-dicarboxylic acid (from the Na salt in H₂O and Th(NO₃)₄); the distillate was then redistd. at 12 mm., converted to semicarbazone in MeOH, which was sepd. and hydrolyzed to the ketone with concd. H₂C₂O₄; it b₁₂ 130.degree.-b₁ 170.degree.. An attempt to prep. 1-methylcyclotridecan-3-one from the Th salt of 2-methyldodecane-1,12-dicarboxylic acid was not successful, yielding a mixt. of products which were not completely sepd. and did not have a musk odor; 1-methylcyclotetradecan-2-one could not be prepd. from the Y salt of tetradecane-1,13-dicarboxylic acid and the product of distn. did not have a ***musk*** odor. 1-Methylcyclotetradecan-4-one, m. 28-9.degree., which has a ***musk*** odor was prepd. by heating to 170-80.degree. the Y salt of 3-methyltridecane-1,13-dicarboxylic acid (133 g.); the 59 g. of product thus obtained was fractionated as follows: (a) 10 g., b₁₂ 65-130.degree.; (b) 4 g., b₁₂ 130-50.degree.; (c) 2.2 g., b₁ 120-40.degree. (d) 5.5 g., b₁ 140-60.degree.; (e) 2.8 g., b₁ 160-80.degree.; all of these fractions were converted to the semicarbazone (d yielding the most), m. 182-3.degree.; from the mother liquors the semicarbazone of Me .omega.-methyltridecyl ketone was obtained, m. 118-9.degree.. An attempt to prep. 1-methylcyclopentadecan-2-one from the Th salt of pentadecane-1,14-dicarboxylic acid was not successful and did not give products with a musk odor. 1-Methylcyclopentadecan-2-one was prepd. by adding 4 g. cyclopentadecanone in 100 cc. abs. Et₂O to 4 g. finely divided NaNH₂ under PhH and cooled with ice, allowed to stand several hrs., then shaken 2 days at room temp. The flocculent Na deriv. of the ketone was decanted with the liquid from the solid residue of NaNH₂, which was washed with abs. Et₂O, the Et₂O suspension boiled 2 days with excess MeI, treated with H₂O and distd., b₁₂ 171-3.degree., converted to semicarbazone (m. 149-50.degree.) and then the ketone was regenerated from the latter with H₂C₂O₄; the odor is similar but slightly less than the unmethylated ketone, d₄₁₆ 0.9213, n_{D16} 1.4812, MD 73.60. 1,3-Dimethylcyclopentadecan-2-one could not be prepd. by distn. of the Y salt of hexadecane-2,15-dicarboxylic acid and

the product of distn. did not have a musk odor. Attempts to prep. 1-methylcyclopentadecan-3-one (dl-musccone) from the Th salt of 2-methyltetradecane-1,14-dicarboxylic acid were not successful but gave a product which is probably Me.omega.-methyltetradecyl ketone, forming a semicarbazone, m. 122.degree.; similarly the Y salt yielded the same semicarbazone (m. 122.degree.) and another semicarbazone which is probably the isomer, m. 83.degree.; regeneration of the ketones from the mother liquors gave an oil with a strong musk odor indicating the presence of traces of dl-musccone. Distn. of the Y salt of 2,13-dimethyltetradecane-1,14-dicarboxylic acid did not yield 1,5-dimethylcyclopentadecan-3-one but gave a very small amt. of Me 2,3-dimethyltetradecyl ketone which was isolated as the semicarbazone, m. 78-9.degree.; the distillate had a very slight musk odor. 1-Methylcyclopentadecan-4-one was prepd. by heating the Th salt of 3-methyltetradecane-1,14-dicarboxylic acid (from 81 g. acid) which yielded 50 g. of distillate and when fractionated gave: (a) 4 g., b12 45-90.degree.; (b) 7.2 g., b12 90-150.degree.; (c) 10.7 g., b0.5 120-60.degree.; (d) 3 g., b0.5 160-80.degree.; (e) 4.2 g., b0.5 180-205.degree.; (f) 8 g., b0.5 205-50.degree.. Fraction c has a decided musk odor and yields the greatest quantity of semicarbazone, m. 161-2.degree., although the latter could be obtained from all fractions; the ketone regenerated from the semicarbazone, b0.5 125.degree. and has an odor which cannot be distinguished from muscone. Distn. of the Th salt of 4-methyltetradecane-1,14-dicarboxylic acid gave 1-methylcyclopentadecan-5-one, which was isolated as the semicarbazone, m. 164.degree.. Methylation of dihydrocibetone gave 1-methylcycloheptadecan-2-one, which b0.5 150.degree. and has an odor that can hardly be distinguished from the unmethylated ketone; the semicarbazone, m. 142-3.degree.. 1-Methylcyclopentadecan-1-ol, m. 85-6.degree., was prepd. by the Grignard reaction from cyclopentadecanone (10 g.) and MeI (5 g.); at the same time a small amt. of compd., m. 162.degree., was obtained and this is probably a dimol. cyclopentadecanone (C30H56O2) or a pinacone (C30H58O2); when the methylcyclopentadecanol was dehydrated by heating several hrs. with 90% HCO2H it gave almost quant. 1-methylcyclo-1-pentadecane, b12 152-3.degree., d422 0.8697, nD22, 1.4853, MD 73.26; reduction with H and Pt black in EtOAc gave methylcyclopentadecane, b12 147-8.degree., d421 0.8576, nD21 1.4735, MD 73.34.

L10 ANSWER 16 OF 16 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1926:14568 CAPLUS

DOCUMENT NUMBER: 20:14568

ORIGINAL REFERENCE NO.: 20:1791i,1792a-f

TITLE: Carbon rings. II. Synthesis of carbocyclic ketones of 10- to 18-membered rings

AUTHOR(S): Ruzicka, L.; Stoll, M.; Schinz, H.

SOURCE: Helvetica Chimica Acta (1926), 9, 249-64

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

ABSTRACT:

The yields of these ketones from di-CO2H acids depend upon the metal used in forming the salt; the decompn. of all of the Th salts of polymethylenedicarboxylic acids investigated yields cyclic ketones. The yield of the 9-C ring ketone was about 1.5%, the 10-C ring ketone 0.1-0.2% with decreasing yields as the no. of carbons in the ring increased until the 18-C ring, where an increased yield was obtained. The cycloheptadecanone is identical with dihydrocivetone (above); the constitution of the polymethylene ketones is established by their oxidation with CrO3 to the normal polymethylenedicarboxylic acids with the same number of C atoms. All of the pure ketones with 12 or more C atoms are solids resembling camphor in appearance; the odor of the ketones with 10 to 12 C atoms is distinctly like that of camphor, the ketone with 13 C atoms has a slight cedar-wood odor as do the concd. forms of those with more C atoms; when dild. the ketones with 14-18 C atoms have a characteristic musk odor which is most noticeable with the 15-C atom ketone. The synthesis of this type of compd. opens the field for the technical prepn. of natural musk and civet odorous principles and shows that the possible no. of C members in a ring is much greater than was

previously considered possible. Cyclodecanone (I), prepd. by treating 196 g. nonane-1,9-dicarboxylic acid in warm EtOH with the calcd. amt. of 20% NaOH, dilg. with H₂O, adding 280 g. ThCl₄, filtering off the sepd. Th salt, drying the 310 g. so obtained at 150.degree., distg. at 12 mm. in 4 portions from a Cu retort at increasing temp. to 500.degree., dissolving the distillate in Et₂O, washing with NaOH and then H₂O, drying over Na₂SO₄ and fractionally distg. at 12 mm.: (a) 50-75.degree. (1.5 g.), (b) 75-90.degree. (2 g.), (c) 90-105.degree. (2.2 g.), (d) 105-20.degree. (1.5 g.), (e) 120-40.degree. (2.6 g.) with 36 g. residue; these fractions in MeOH were allowed to react at room temp. with semicarbazide acetate and the solns. evapd. at room temp.; fractions c and d contained the most (about 20 mg.) semicarbazone (II), m. 200-1.degree.; when II is hydrolyzed with hot H₂C₂O₄ soln., extd. with Et₂O, washed with Na₂CO₃ and distd. in vacuo, I is obtained as an oil, b₁₂ 100-2.degree.. Oxidation of I in AcOH with CrO₃ gave sebacic acid. The following cyclic ketones were obtained like I from the corresponding di-CO₂H acids with one more C atom; each cyclic ketone on oxidation gave a di-CO₂H acid with the same no. of C atoms: cyclo-undecanone, oil, b₁₂ 110.degree.; semicarbazone, m. 200.degree.; cyclododecanone, m. 59.degree., b₁₂ 126-8.degree.; semicarbazone, m. 220.degree.; cyclotridecanone, m. 32.degree., b₁₂ 137-9.degree.; semicarbazone, m. 200.degree.; cyclotetradecanone m. 52.degree., b₁₂ 155-6.degree.; semicarbazone, m. 195.degree.; cyclopentadecanone or "exaltone," m. 63.degree., b_{0.3} 120.degree.; semicarbazone, m. 180.degree.; cyclohexadecanone, m. 56.degree., b_{0.5} 138.degree.; semicarbazone, m. 180.degree.; cycloheptadecanone, or dihydrocivetone, m. 63.degree., b_{0.3}, 145.degree.; semicarbazone, m. 191.degree.; cyclooctadecanone, m. 71.degree., b_{0.3} 157-9.degree..

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COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
77.35	225.92

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
-11.07	-11.07

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